

DECONTAMINATING SOLAR WIND SAMPLES WITH THE GENESIS ULTRA-PURE WATER MEGASONIC WAFER SPIN CLEANER. M.J. Calaway¹, M.C. Rodriguez², J.H. Allton³, and E.K. Stansbery³: (1) Jacobs (ESCG) at NASA Johnson Space Center, Houston, TX; (2) Geocontrol Systems (ESCG) at NASA Johnson Space Center, Houston, TX; (3) NASA, Johnson Space Center, Houston, TX; michael.calaway-1@nasa.gov.

Introduction: The Genesis sample return capsule, though broken during the landing impact, contained most of the shattered ultra-pure solar wind collectors comprised of silicon and other semiconductor wafers materials. Post-flight analysis revealed that all wafer fragments were littered with surface particle contamination from spacecraft debris as well as soil from the impact site. This particulate contamination interferes with some analyses of solar wind. In early 2005, the Genesis science team decided to investigate methods for removing the surface particle contamination prior to solar wind analysis [1].

Previous JSC Techniques: The NASA JSC Genesis curation team began to develop first order semiconductor wafer cleaning strategies using in-house manufactured ultra-pure water (UPW) coupled with a Honda Electronic W-357LS-80 megasonic cleaner in an ISO 4 (class 10) cleanroom [2]. This cleaner was originally used prior to flight to clean Genesis flight hardware during science canister payload assemblage in 1999 to 2001 [3]. The typical properties of UPW used in JSC curatorial laboratories consist of $18\text{ M}\Omega$ resistivity, ionic levels in the low ppt and TOC $< 10\text{ ppb}$. The W-357LS-80 megasonic cleaner was able to clean wafers. However, the smaller fragments that ranged from 1 to 15 mm did not clean well under an 8.0 l/min. UPW flow.

In 2006, a Honda Electronic W-357P-25 megasonic pulse jet cleaner operating at 1 MHz oscillation at 60 W (0.4 A) with an 1.5 liters/min. flow rate mounted onto a wet bench provided a more focused flow of UPW [4]. However, this technique also had several disadvantages. First, the cleaning process required manually holding each wafer with tweezers for several minutes. The manual holding of the wafer created a risk of falling into the wet bench and damaging the wafer. The sonicated UPW was also not very well dispersed over the entire wafer. Several experiments also revealed that 1 to 5 micron size particles would be greatly reduced by moving the wafer around. In addition, more particles were removed with longer UPW exposure times that could not be accomplished with manually holding the wafer.

New UPW/megasonic Technique: From 2006-2007, the Genesis Ultra-pure Water Megasonic Wafer Spin Cleaner (fig. 1) was developed to mitigate and solve some of the problems associated with standard UPW/megasonic cleaning.

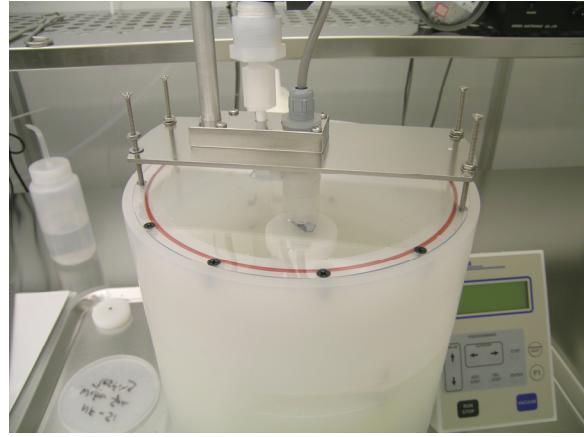


Fig. 1: Genesis Ultra-pure Water Megasonic Wafer Spin Cleaner.

We integrated two existing technologies: a Laurell Technologies Corp. WS-400E-NPP-Lite Series Natural Polypropylene Single Wafer Spin Processor and the W-357P-25 megasonic cleaner. The spin processor would replace manual holding of the sample with a vacuum mount that would hold various sizes of irregular shaped wafer fragments 3 mm to 10 cm in diameter. The processor also had the capabilities to spin the wafer at 1000 to 10000 RPM. We also designed and fabricated a holder for the W-357P-25 megasonic pulse jet cleaner and placed this devise in the modified lid of Laurell's single wafer spin processor. The UPW/megasonic nozzle was aligned such that it was directly in the center of the spinner at 3-5 mm distance from the wafer surface. Several non-flight and flown silicon wafer cleaning tests confirmed that Genesis wafer fragments could be decontaminated without damaging the implanted solar wind.

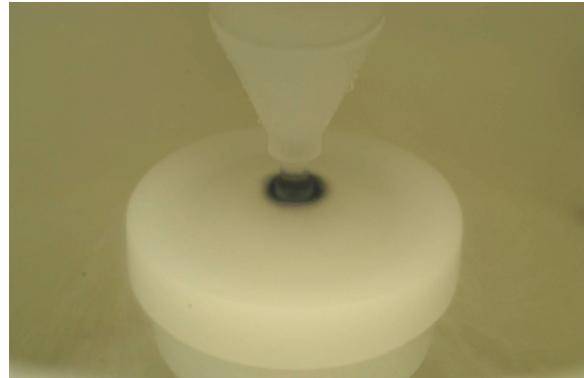


Fig. 2: UPW/megasonic cleaning of a Genesis Si fragment at 3000 RPM.

Cleaning Efficiency Experiments: The effectiveness of this first order cleaning technique for all Genesis array fragments was studied by measuring particles from images before and after UPW cleaning. For this experiment, the UPW megasonic wafer spin cleaner was set with a 1.5 l/min. UPW flow at 40° C, 0.4 A sonication, and 3000 RPM spin. Si sample cleaning times then varied between 0, 5, 15, and 30 min. A $\sim 1 \text{ mm}^2$ area was mosaic imaged with a Leica DM6000 M optical microscope with a 50X lens at all cleaning times to compare particle counts from images using ImageProPlus software.

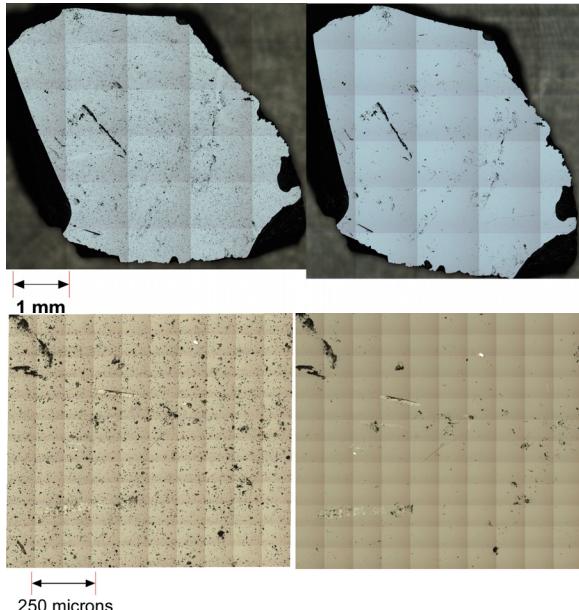


Fig. 3: Mosaic images of Genesis Si sample 60458 before and after UPW/megasonic cleaning.

Results: Preliminary results from cleaning flown Genesis Si sample 60458 shows that the Genesis Ultra-pure Water Megasonic Wafer Spin Cleaner reliably removes the majority of surface particle contamination (fig. 3). The remaining surface features are impact gouges, scratches, highly statically charged particles and material chemically bonded to the surface. Fig. 4 and 5 shows the resulting particle counts from Si sample 60458 with an 87 to 90% particle reduction. The contamination free surface area also increased from 93.2% to 98.6%. The area measurement shows that the most efficient cleaning time for Genesis Si samples is 15 min.

Previous UPW megasonic cleaning studies on Genesis Si sample 60178 in 2006 without the spin processor method removed 51% particles below 1 micron [4].

Number of Particles	Clean Time			
	0 min.	5 min.	15 min.	30 min.
>30 μm diameter	492	6	7	7
10-30 μm diameter	1145	21	17	17
5-10 μm diameter	1869	86	78	89
1-5 μm diameter	13379	1187	1470	1237
0.3-1.0 μm diameter	10697	1400	2114	1600
Total Particle Count	27582	2700	3686	2950
% Particle Reduction		90.21%	86.64%	89.30%
Total Area of Particles (μm^2)	74537.55	16570.56	15504.54	15501.39
Surface Area Free of Contamination	93.20%	98.49%	98.59%	98.59%

Fig. 4: Si Sample 60458 cleaning results.

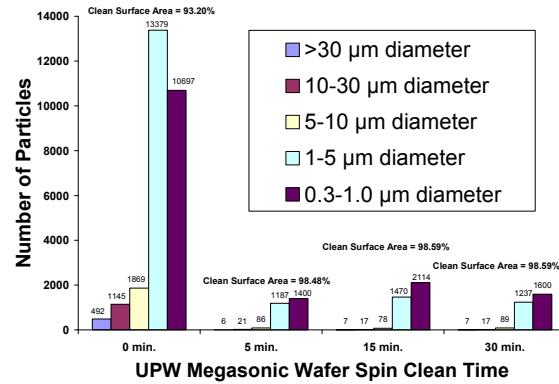


Fig. 5: Si Sample 60458 cleaning results.

On 60458, the Genesis Ultra-pure Water Megasonic Wafer Spin Cleaner removed 87% of particles below 1 micron after 5 min. at 3000 RPM. Therefore, preliminary results show that the Genesis Ultra-pure Water Megasonic Wafer Spin Cleaner technique has better efficiency than previous methods and should be used for a first order particle decontamination before other cleaning methods are applied to remove the remaining contamination.

More cleaning efficiency tests are required to obtain enough statistics to validate this wafer cleaning technique. Further work is also required to characterize the surface of the Si sample 60458 with SEM/EDS analysis. Additional SEM/EDS analysis can help determine the type of contamination that is not being removed. Cross-sectional stratigraphic profiles of particle contamination excavated by FIB and analyzed by TEM are also planned to help characterize the surface interaction between the remaining contamination and wafer surface.

References: [1] Lauer, H.V. et. al. (2005) LPSC XXXVI, Abstract # 2407. [2] Allton, J.H. et. al. (2006) LPSC XXXVII, Abstract # 2324. [3] Stansbery, E.K. et. al. (2001) LPSC XXXII, Abstract # 2084. [4] Allton, J.H. et. al. (2007) LPSC XXXVIII, Abstract # 2138.